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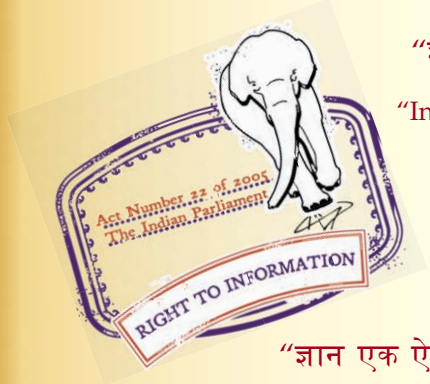
IS 11540 (1986): Michler's Ketone [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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IS : 11540 - 1986
(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR MICHLER'S KETONE

UDC 667·281 : 547·631·6'415·1

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

June 1986

Indian Standard

SPECIFICATION FOR MICHLER'S KETONE

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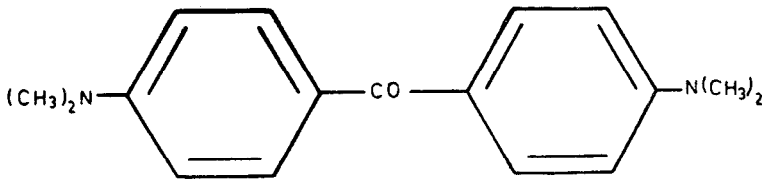
Indian Standard

SPECIFICATION FOR MICHLER'S KETONE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 January 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Michler's ketone ($C_{17}H_{20}ON_2$) is an important dye intermediate used in the manufacture or triphenyl methane dyes. It is also known as 4, 4'-tetramethyl diaminobenzophenone and it is represented by the following structural formula:



Michler's Ketone
(Molecular Mass 268)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for Michler's ketone.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of green to blue coloured powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR MICHLER'S KETONE

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			IS : 5299-1969*	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, *Max*	0·5	9	—
ii)	Ash, percent by mass, *Max*	0·5	11	—
iii)	Matter insoluble in methanol, percent by mass, *Max*	0·5	10	—
iv)	Melting point, °C, *Min*	170	8	—
v)	Crystal violet, percent by mass, *Max*	0·5	—	A

*Methods of sampling and tests for dye intermediates.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Tare, net and gross mass; and
d) Batch number.

*Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Tests for the determination of melting point, moisture, matter insoluble in methanol, ash and crystal violet shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 For declaring the conformity of the lot to the requirements of all characteristics tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **2.1** and Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.
†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF TEST FOR MICHLER'S KETONE

A-1. DETERMINATION OF CRYSTAL VIOLET

A-1.0 General — Crystal violet in Michler's ketone is estimated by thin layer chromatography.

A-1.1 Apparatus

A-1.1.1 *Chromatographic Chamber* — 22 × 10 × 22 cm.

A-1.1.2 *Micropipette* — 10 µl capacity.

A-1.1.3 *Thin-layer Chromatographic Glass Plates* — 20 × 20 cm.

A-1.1.4 *Adjustable Thin Plate Applicator*

A-1.1.5 *Chromatographic Spray Bottle*

A-1.1.6 *Weighing Bottle*

A-1.1.7 *Oven*

A-1.2 Reagents

A-1.2.1 *Sample Solution* — 1 percent solution of Michler's ketone in dimethylformamide : Actone : Ammonia (10:80:10).

A-1.2.2 *Reference Solution* — 0·01 percent solution of crystal violet in dimethylformamide : Acetone : Ammonia (strong) (10:80:10).

A-1.2.3 *Developer*

a) Toluene : Methanol (95 : 5)

b) Glacial Acetic Acid : Methanol (99:1)

A-1.3 Procedure

A-1.3.1 *Preparation of Chromatographic Plates* — Mix 50 g of neutral silica gel *G* with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel *G* layer of thickness not exceeding 0·25 mm, and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel *G* from either of the lateral sides of each plate.

A-1.3.2 *Chromatographic Separation* — Pipette out 10 μl of the sample solution (**A-1.2.1**) and 5 μl of reference solution (**A-1.2.2**) in the form of a uniform line of about 14 cm length on the plate, at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the developer (a) (**A-1.2.3**) to run up to 15 cm at room temperature. Take out the plate and dry completely. Next allow the developer (b) (**A-1.2.3**) to run up to 15 cm at room temperature. Take out the plate and dry completely.

A-1.3.3 *Evaluation* — By visual comparison, the spot corresponding to crystal violet in the sample shall have an equal or less intensity than that of the reference sample corresponding to 0·5 percent of crystal violet.